IS 4006-3 (1985): Methods of test for paper and pulp based packaging materials: Part III [CHD 15: Paper and its products]

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( Reaffirmed 2002 )

# *Indian Standard*

# METHODS OF TEST FOR PAPER AND PULP BASED PACKAGING MATERIALS, PART 3

## *( First Revision )*

UDC 676·24·01 : 621·798·156



INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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# *Indian Standard*

# METHODS OF TEST FOR PAPER AND PULP BASED PACKAGING MATERIALS, PART 3

## *( First Revision )*

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# *Indian Standard*

# METHODS OF TEST FOR PAPER AND PULP BASED PACKAGING MATERIALS, PART 3

# *( First Revision )*

## 0. FOREWORD

**0.1** This Indian Standard ( First Revision ) was adopted by the Indian Standards Institution on 30 July 1985, after the draft finalized by the Paper and Pulp Based Packaging Materials Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard is the third part in the series of standard for methods of tests for paper and pulp based packaging materials.

**0.3** The packaging materials used in industry are many and varied. They are paper and paper products, textiles, metal and metal foils, plastics and a variety of laminates, wood, glass and ceramics, cushioning materials, strapping and hooping materials, nails, etc. Among these, paper and paper products are of major importance.

**0.4** This standard was first published in 1978. The committee decided to revise the standard to incorporate spectrophotometric methods also for the determination of copper and iron which are faster and accurate.

**0.5** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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## 1. SCOPE

**1.1** This standard ( Part 3 ) prescribes methods of test for the determination of the following in paper and pulp based packaging materials:

a) Arsenic content,

b) Total copper content,

*Rules for rounding off numerical values ( *revised* ).

c) Total iron content,

d) Water soluble copper content, and

e) Water soluble iron content.

**1.2** Should any inconsistency exist between the requirements of this standard and those of the standard for an individual material, the latter shall prevail.

## 2. TERMINOLOGY

**2.1** For the purpose of this standard, the definitions given in IS : 4261-1967* shall apply.

## 3. SAMPLING

**3.1** Representative samples for test shall be drawn as prescribed in 3 of IS : 1060 ( Part 1 )-1966†.

## 4. QUALITY OF REAGENTS

**4.1** Unless otherwise specified, pure chemicals and distilled water ( *see* IS : 1070-1977‡ ) freshly boiled and cooled, shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## 5. ARSENIC CONTENT

### 5.1 Procedure

**5.1.1** Weigh 5 g of the sample, cut into strips, fold and place in a Kjeldahl flask, which shall be used only for this purpose. Add 20 ml of sulphuric acid. Warm very gently till the initial reaction is over and then more strongly, adding nitric acid, 1 ml at a time, whenever white fumes indicate that an excess of nitric acid is no longer present. Care shall be taken that fuming of the acid does not start until all the organic matter is destroyed. Continue till the paper is completely oxidized. When further addition of nitric acid produces no further change in the yellow or green solution, cool, carefully dilute to 50 ml with water and re-evaporate until copious white fumes are evolved. Repeat the operation of dilution and evaporation to ensure that the nitric acid has been entirely removed.

*Glossary of terms relating to paper and pulp based packaging materials.
†Methods of sampling and test for paper and allied products: Part 1 ( *revised* ).
‡Specification for water for general laboratory use ( *second revision* ).

**5.1.2** Determine the arsenic content, using the working solution prepared in **5.1.1**, as prescribed in IS : 2088-1983*.

**5.1.3** In case of dispute the spectrophotometric method using silver diethyldithiocarbamate as prescribed in IS : 2088-1983*, shall be used as the referee method for determination of arsenic content.

## 6. DETERMINATION OF TOTAL COPPER

**6.0 General** — Two methods have been prescribed for the determination of total copper. Method A is the routine method and Method B is the referee method.

### 6.1 Method A

**6.1.1** *Reagents*

**6.1.1.1** *Citric acid*

**6.1.1.2** *Ammonium hydroxide solution* — relative density 0·90.

**6.1.1.3** *Sodium diethyldithiocarbamate* — 0·2 percent solution, freshly prepared.

**6.1.1.4** *Standard copper solution* — 1 ml = 0·01 mg of copper.

**6.1.2** *Procedure*

**6.1.2.1** Prepare a working solution by wet ashing 5 g of the sample as specified in **5.1.1** for determination of arsenic. Dilute to 100 ml.

**6.1.2.2** Take 20 ml or other suitable aliquot of the working solution and add 1 g of citric acid and one or two drops of thymol blue indicator. Make alkaline with ammonia solution. Make up to 50 ml in a Nessler cylinder. Add 5 ml of 0·2 percent solution of sodium diethyldithiocarbamate. Match any yellow colour produced against a standard solution of copper which has been treated in the same way.

Make a blank test on the reagents.

**6.2 Method B** — Determine copper by the spectrophotometric method as prescribed in **7** of IS : 7212-1974†.

## 7. DETERMINATION OF TOTAL IRON

**7.0 General** — Two methods have been prescribed for the determination of total iron. Method A is the routine method and Method B is the alternate referee method.

*Methods for determination of arsenic ( *second revision* ).
†Methods of determination of copper.

**7.1 Method A**

**7.1.1** *Reagents*

**7.1.1.1** *Hydrochloric acid* — 20 percent *v/v*.

**7.1.1.2** *Thioglycollic acid* — 5 percent solution.

**7.1.1.3** *Ammonium hydroxide solution* — relative density 0·90.

**7.1.1.4** *Standard iron solution* — Prepared from ammonium ferrous sulphate oxidized with potassium permanganate solution and hydrochloric acid ( 1 ml = 0·005 mg of iron ).

**7.1.2** *Procedure* — Ash 5 g of the sample in a platinum dish at a temperature not exceeding 600°C. Evaporate the residue with 2 ml of 20 percent hydrochloric acid on a water-bath. Make up to 100 ml in a measuring flask. Transfer 25 ml or other suitable aliquot to a 50-ml Nessler cylinder. Add 5 ml of 5 percent aqueous solution of thioglycollic acid; make alkaline with ammonia solution. Dilute to 50 ml with water. Allow to stand for 5 minutes. Match the resulting colour against a series of standard iron solutions treated in the same way. Make a blank test on the reagents used.

**7.2 Method B** — Determine iron by the spectrophotometric method as prescribed in **5** of IS : 4542-1968*.

## 8. DETERMINATION OF WATER-SOLUBLE COPPER

**8.1 Reagents**

**8.1.1** *Sulphuric Acid* — relative density 1·84.

**8.1.2** *Sodium Nitrate*

**8.1.3** *Sulphuric Acid* — 20 percent *v/v*.

**8.2 Preparation of Working Solution**

**8.2.1** Weigh exactly 5 g of the sample and cut into strips about 2 cm wide and of any convenient length. Fold these in zig-zag fashion so as to have a little bundle 2 cm high when arranged on edge side by side in a 250-ml beaker, thus preventing packing but exposing a maximum stable surface to the action of the solvent, which is distilled water. Extract with 4 successive 100 ml portions each for 30 minutes at 85 to 95°C to give a total volume of 400 ml and taking a total time of 2 hours.

*Colorimetric method for determination of iron.

**8.2.2** Filter each washing immediately through a filter using a sintered glass filter. This washing can be rapidly evaporated in a weighed flat bottomed dish of platinum, porcelain, or silica, while the next extraction proceeds.

**8.2.3** Evaporate to dryness on a water-bath and heat for 2 hours in an air-oven at 105 ± 1°C.

**8.2.4** Dissolve in hot water free from iron and copper, and transfer to a 50-ml beaker. Evaporate to dryness and char gently. Add 20 ml of concentrated sulphuric acid and heat carefully until fuming strongly, then add sodium nitrate gradually until the last traces of carbonaceous matter have been cleared away. About 2 g of sodium nitrate will normally be needed. When perfectly clear, cautiously evaporate the acid to dryness, the mineral residue being allowed to cool. Take with 10 ml of 20 percent sulphuric acid, warm and dilute with water making up to 50 ml of working solution.

NOTE — This method of preparation of the sample is necessary in order to avoid loss of volatile water-soluble copper and iron salts. Preparation of sample by the more obvious method of 'ashing' tends to give a low result.

**8.3 Procedure** — Take 20 ml or other suitable aliquot of the working solution and proceed as described in **6.1.**

**8.4** Alternatively, the spectrophotometric method as prescribed in **7** of IS : 7212-1974* may be followed.

## 9. DETERMINATION OF WATER-SOLUBLE IRON

**9.1** Take 5 ml or other suitable aliquot of the working solution prepared in **8.2.4** in 50 ml Nessler cylinder and proceed as described in **7.1.**

**9.2** Alternatively, the method prescribed as in **5** of IS : 4542-1968† may be followed.

*Methods of determination of copper.
†Colorimetric methods for determination of iron.

# INTERNATIONAL SYSTEM OF UNITS ( SI UNITS )

**Base Units**

| Quantity | Unit | Symbol |
|---|---|---|
| Length | metre | m |
| Mass | kilogram | kg |
| Time | second | s |
| Electric current | ampere | A |
| Thermodynamic temperature | kelvin | K |
| Luminous intensity | candela | cd |
| Amount of substance | mole | mol |

**Supplementary Units**

| Quantity | Unit | Symbol |
|---|---|---|
| Plane angle | radian | rad |
| Solid angle | steradian | sr |

**Derived Units**

| Quantity | Unit | Symbol | Definition |
|---|---|---|---|
| Force | newton | N | 1 N = 1 kg.m/s$^2$ |
| Energy | joule | J | 1 J = 1 N.m |
| Power | watt | W | 1 W = 1 J/s |
| Flux | weber | Wb | 1 Wb = 1 V.s |
| Flux density | tesla | T | 1 T = 1 Wb/m$^2$ |
| Frequency | hertz | Hz | 1 Hz = 1 c/s (s$^{-1}$) |
| Electric conductance | siemens | S | 1 S = 1 A/V |
| Electromotive force | volt | V | 1 V = 1 W/A |
| Pressure, stress | pascal | Pa | 1 Pa = 1 N/m$^2$ |

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